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Dynamic packing of ion-exchange chromatographic columns*

The conventional method of packing ion-exchange resin into a chromatographic column is to make a slurry of the resin beads and introduce it into the column or a chamber attached to the column, and then allow the resin particles to settle by gravity to form the packed bed (for example, ref. 1). Liquid supernate is removed when necessary and additional slurry added until the column is full of resin.

This technique has a disadvantage when the sizes of the resin particles vary over a large range, because the larger particles will settle more rapidly and the resulting bed will have longitudinal zones of different particle sizes. If the column is not perfectly vertical during gravitational packing, there can also be radial variation in particle size. These variations affect the resolution of the column, and it will certainly make it difficult to pack several columns with reproducible properties.

An additional problem exists when very small resin particles (less than 20 μ in diameter) are used in the chromatographic column. When the gravitational settling method is used for such particles, the settling velocity is very low and the time required to pack a column is prohibitive.

To circumvent both of these problems, we use a technique of dynamic packing in which the ion-exchange particles are forced into the packed bed in a flowing fluid at a velocity much greater than their settling velocity.

Experimental technique

Chromatographic columns using ion-exchange resin can be packed dynamically either by displacing a thick slurry or by extruding a prepacked bed.

Packing with a slurry. Dynamic packing with a slurry is accomplished by connecting a chamber or reservoir to the chromatographic column, filling the chamber with a thick slurry of the ion-exchange resin, and then displacing the slurry into the column with a liquid that is pumped into the top of the slurry chamber (see Fig. 1). If the linear velocity of the displacement fluid in the slurry chamber is substantially greater than the settling velocity of the largest particle, there will not be size segregation in the resulting packed bed.

A slurry of 25--50 vol.% solids is used; and, in most cases, the slurry chamber is sufficiently large to hold enough slurry to pack the column without refilling. In the event that the volume of the column is larger than the volume of the slurry chamber, the following procedure is used: (1) a chamber volume of slurry is packed into the column; (2) the supernatant liquid in the slurry chamber is removed by siphoning; (3) another batch of slurry is added; and (4) the previous three steps are repeated as needed. This technique has been used to pack high-pressure anion-exchange columns with 5 to 10 μ diameter particles[2,3]. Such columns that are 0.62 cm diameter $\times$ 150 cm long can be packed in about 1 h.

Best results are obtained when the column is first filled with clear liquid before the slurry chamber is attached. Then, when the slurry is forced into the column, air is excluded and there are no gross turbulences that can cause mixing problems.

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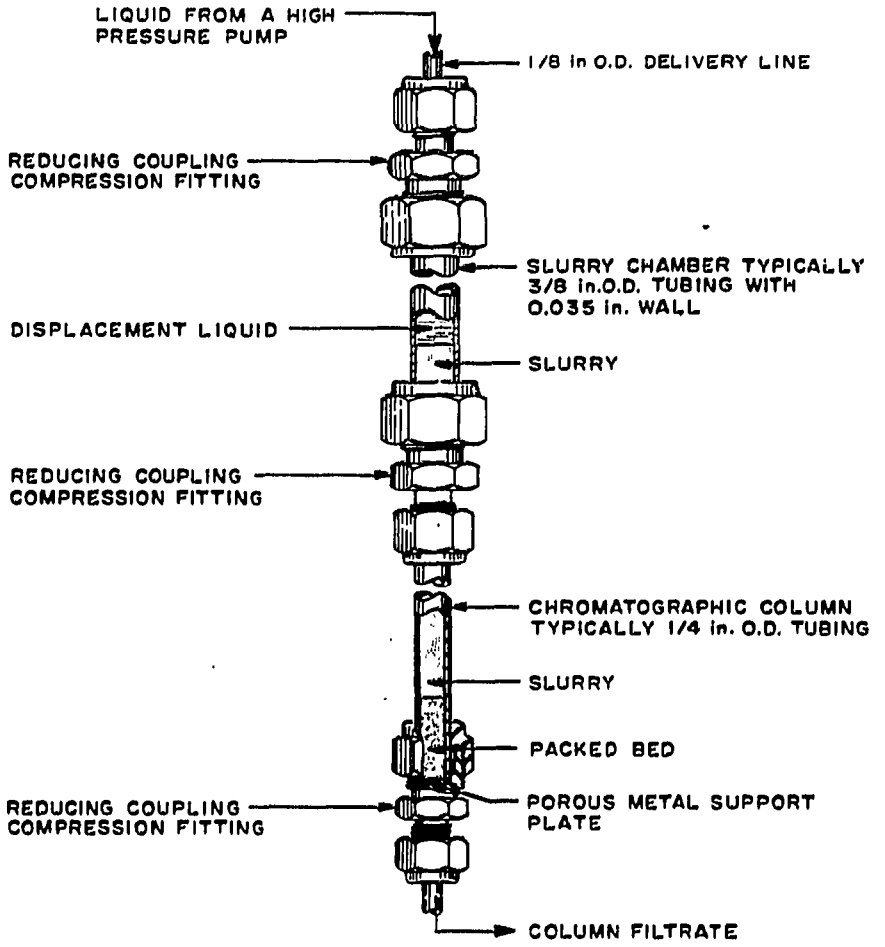

Fig. 1. Experimental set-up for dynamic packing of a high-pressure chromatographic column.

In all of our work aqueous buffer solutions were used for elution. The liquid used in packing was the buffer solution whose concentration results in the smallest amount of resin bead swelling and, hence, in the packed bed of highest density.

Packing by extrusion. In packing small diameter columns of appreciable length (0.45 cm diameter × 200 cm long column[4]) with small ion-exchange resin particles (5 to 10 μ diameter), we have found it difficult to pump a liquid through the slurry chamber and into the column at a sufficiently rapid rate to prevent size segregation. This difficulty is caused by a high pressure drop (in the long column), which prevents rapid displacement of the slurry from the slurry chamber into the column. However, as an alternative, one can first pack a fixed bed into a reservoir (or cartridge) of larger diameter and then extrude that bed into the small diameter chromatographic column by displacing it with the liquid.

This extrusion technique can also be used to pack a coiled column with assurance of constant packing properties and no void spaces throughout the column. In addition, it has also proved very useful for preparing column charges to be transported from our laboratory to other facilities. It is possible that such a method could be exploited by resin manufacturers as a means of providing new column charges to customers with minimal customer effort in packing the column.

For extrusion packing, the chromatographic column should be dry at the beginning so that the extruded bed will not be diluted. However, the packed bed in the cartridge must be kept moist to prevent bed caking. Very high pressures are required for extrusion when caking occurs. Because of this problem, we have been unsuccessful in transferring ground resin (irregular shapes) by extrusion; however, spherical resin particles down to the size of 3 to 7 μ in diameter have been successfully packed.

Experimental results

To compare dynamic packing with the gravitational settling methods, two 0.62 cm diameter × 50 cm long columns were packed with nominal 10 to 20 μ diameter anion-exchange resin in distilled water, using each of the two techniques. The resin beds were then removed by extrusion, and the particle size distribution in sections of each bed at different positions was determined by photomicroscopy. As seen in Table I, the dynamically packed column had essentially uniform particle size distribution throughout the column, whereas the column packed by settling had a much higher proportion of large particles in the bottom of the column and a gradation to much smaller particles at the top of the column.

TABLE I

SIZE DISTRIBUTION OF PARTICLES IN CHROMATOGRAPHIC COLUMNS THAT WERE PACKED BY DYNAMIC METHODS AND BY GRAVITATIONAL SETTLING[a]

Particle size distribution for initial feed: average particle size, 17.9 μ diameter; standard deviation, 5.9 μ.

Height from bottom of column (cm)	*Dynamically packed*[b]		*Gravitational settling*	
	Average particle size (μ)	*Standard deviation (μ)*	*Average particle size (μ)*	*Standard deviation (μ)*
0	18.3	5.0	21.6	5.6
10	18.2	5.6	20.1	5.7
20	17.7	6.3	19.7	5.4
30	18.1	5.6	18.5	5.8
40	18.0	6.0	17.0	5.7
50	17.8	6.2	14.5	4.6

[a] A 0.62 cm diameter × 50 cm long column was loaded from a 0.78 cm diameter × 75 cm long slurry reservoir. One reservoir charge of approximately 50 vol.% resin was used. Each particle size distribution was based on the microscopic measurement of 100 particles.

[b] The fluid velocity in the slurry reservoir during loading was approximately ten times the settling velocity of a 20 μ particle.

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